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ATOFINA

ATOFINA Chemicals, Inc.

March 5, 2002

UPS NEXT DAY DELIVERY

Document Control Office (TS-7407M)
Attn: TSCA Section 8(e) Coordinator
Office of Pollution Prevention and Toxics
U.S. Environmental Protection Agency
1201 Constitution Avenue, N.W.
Washington, DC 20460

Contain NO CBI

Subject: TSCA 8(e) Submission

Dear Sir/Madam:

The European Peroxide Producers Group is submitting the enclosed acute aquatic toxicity to *Daphnia* test to the Environmental Protection Agency (EPA) pursuant to the Toxic Substances Control Act (TSCA) Section 8(e). The study provides information on neodecaneperoxoic acid, 1,1-dimethylethyl ester, (CAS No. 26748-41-4) and does not involve effects in humans.

This submission is being made by ATOFINA Chemicals, Inc. on behalf of the European Peroxide Producers Group member companies: AKZO-NOBEL Polymer Chemicals LLC, ATOFINA, and Peroxid-Chemie Degussa. Nothing in this letter or the enclosed test report is considered confidential business information of the European Peroxide Producers Group or of the sponsoring companies in the Group.

The concentration of the water accommodated fraction of test substance that immobilized 50% of the *Daphnia magna* (EC_{50}) was calculated to be 0.79 mg/l.

Further questions regarding this submission may be directed to me at (215) 419-5890.

Best Regards,

Debra Randall

Debra Randall, D.A.B.T.
Product Safety Manager

ATOFINA Chemicals, Inc.
2000 Market Street
Philadelphia, PA 19103-3222
215-419-7000
www.AtofinaChemicals.com

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Chemicals Research
Arnhem

Final Research Report

CGS-ENV F01034 T 01001 ODA August 8, 2001

M.G.J. Geurts, I.C.M. Garttener-Arends

ACUTE TOXICITY OF THE WATER ACCOMODATED FRACTION OF TERT-BUTYLPEROXYNEODECANOATE IN SOLVENT TO *DAPHNIA MAGNA*

Project identification

Clients	Akzo Nobel Polymer Chemicals, Laporte GmbH, Atofina	Client No	Project No
Subclient		Subclient No	Task No 68996

Drafted by department Environmental Chemistry

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ABSTRACT

The acute toxicity of the Water Accommodated Fraction (WAF) of Tert-Butyl Peroxyneodecanoate to *Daphnia magna* (water flea) was tested under static conditions for 48 hours in accordance with OECD and EEC guidelines for testing of chemicals, and ECETOC, Monograph 26(1996). The test was performed in compliance with Good Laboratory Practice (GLP).

Tert-Butyl Peroxyneodecanoate in solvent is immiscible with water and therefore a WAF was prepared. The following dilutions of the WAF were tested: 1:16 – 1:8 – 1:4 – 1:2 and the undiluted WAF. Chemical analyses of the control and WAF were performed and showed that the test substance is probably unstable under test conditions. At the beginning of the test a concentration of 3.05 mg/l was measured. At the end of the test a concentration below 1.64 mg/l was measured. The concentration at the beginning of the test was used for the calculation of the effect concentrations.

The EC_{50} (48 h) was calculated to be 0.79 mg/l with 95% confidence limits of 0.68 and 0.93 mg/l.

The highest concentration causing no immobility (no observed effect concentration, NOEC) after 48 hours amounted to 0.38 mg/l, whereas 0.76 mg/l was the lowest tested concentration (LOEC,48 h) at which significant immobility was observed as compared to the control. The EC_{100} was 1.53 mg/l, which was observed after 48 hours.

Sublethal effects were not observed in the test concentrations where no immobility was observed.

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ACUTE TOXICITY OF TERT-BUTYLPEROXYNEODECANOATE TO *DAPHNIA MAGNA*

Sponsor	Akzo Nobel Polymer Chemicals P.O. Box 247 3800 AE Amersfoort The Netherlands Laporte GmbH Dr. GustavsonAdolphstrasse 3 82049 Pullach Germany Atofina Cours Michelet La Defense 10 92091 Paris France
Study monitor	Ir. W. Cious

STUDY ORGANISATION

Location	Akzo Nobel Chemicals Research Environmental Chemistry Department Velperweg 76 6824 BM Arnhem The Netherlands
Study director	Ing. M.G.J. Geurts
Quality Assurance Unit	Ir. J.M. Plantinga
Management, Head of Department CGS	Dr.Ir. C.J. Groenenboom
Initiation date of the study	11-6-2001
Completion date of the study	26-6-2001

ARCHIVING AND STORAGE

The project file including the final report, amendments to the final report, the study plan, amendments to the study plan, records of quality assurance inspections, all letters, memos and notes and raw data pertaining to the study will be retained in the archives of Akzo Nobel Chemicals Research Arnhem for a period of ten years. Other records including master schedule sheet, laboratory notebooks, logbooks, records of the maintenance and calibration of equipment, summary of training, curricula vitae and job descriptions of the personnel involved in the study, records related to location and storage of the test substance will also be kept in the Akzo Nobel Chemicals Research Arnhem archives for a period of ten years. Test material will be stored deepfrozen under the sample code T 01001 for ten years or only as long as the quality of the test substance permits evaluation.

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GLP COMPLIANCE STATEMENT

The study reported here was carried out according to the study plan in compliance with the OECD Principles of Good Laboratory Practice. The report contains an accurate description of the results.

Study director
Ing. M.G.J. Geurts

date 9-08-2001

Management, Head of Department CGS
Dr.Ir. C.J. Groenenboom

date 13-8-2001

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QUALITY ASSURANCE STATEMENT

This report was audited by the Quality Assurance Unit of Akzo Nobel Chemicals Research Arnhem. It is considered to be an accurate presentation of the methods and procedures applied in the course of the study and an accurate reproduction of the data recorded.
Listed below are the dates of inspection of this study by the Quality Assurance Unit and the dates on which its findings were reported to Study Director and Management.

Dates of inspection	Dates of reporting
13-06-2001	18-06-2001
05-07-2001	05-07-2001

Quality Assurance Unit
Ir. J.M. Plantinga

date 16-8-2001

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CONTENTS

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1. INTRODUCTION

The purpose of this study was to determine the acute toxicity of the Water Accommodated Fraction of Tert-Butylperoxyneodecanoate in solvent to *Daphnia magna* (water fleas) under continuous exposure during 48 hours in a static test.

Acute toxicity is the discernible adverse effect induced in an organism within a short time of exposure to a substance.

The acute toxicity of a test substance is expressed as the median effective concentration (EC_{50}), which is the concentration in water, which immobilizes 50% of the test animals within a period of continuous exposure. Those animals, which were not able to swim for 15 seconds after gentle agitation of the test vessel, were considered to be immobile.

Other endpoints, which will be, derived from the results when possible are the highest tested concentration causing no immobilization (NOEC) and the lowest tested concentration with significant immobility as compared to the control (LOEC) was determined and the lowest concentration causing 100% (EC_{100}) immobility. The swimming ability and other sublethal effects, such as being trapped at the water surface, were recorded after 24 and 48 hours, if observed.

2. TEST GUIDELINES, MODIFICATIONS AND DEVIATIONS

The test was carried out in accordance with OECD (8.1) and EEC (8.2) guidelines for testing of chemicals with the following deviation:

- The test was carried out with the water accommodated fraction prepared from 0.5 g/l test substance.
- The actual test temperature of the test room varied less than 2°C instead of less than 1°C.

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3. MATERIALS

3.1 Test substance

A sample of tert-butylperoxyneodecanoate in solvent (project sample code T 01001) was received on 16-01-2001. The following test substance data were submitted by the sponsor, who accepted full responsibility for the validity thereof.

Akzo Nobel trade name	tert-butylperoxyneodcanoate in solvent
Composition	Tert-butylperoxyneodecanoate 75%; CAS-reg. No. 26748-41-4
	Isododecane 25%; CAS-reg. No. 31807-53-3
Batch number	0010130676
Appearance	Clear liquid
Water solubility	Immiscible
Stability	assumed to be stable at test and storage conditions
Storage until required	freezer

For additional information on the compound see Annex 1 for the certificate of analysis

3.2 Chemicals

All reagents used were of reagent grade quality and obtained from J.T. Baker Chemicals BV, Deventer, The Netherlands, Janssen Chimica, Tilburg, The Netherlands and Fluka chemica, Buchs, Switserland.

3.3 Deionized water

The deionized water used contained not more than 0.01 mg/l of cooper, had a TOC-content of not more than 2.0 mg/l and a conductivity of less than 5 µS/cm. This water was produced from tap water in a water purification system according to Standard Operation Procedure K 10 (8.3).

3.4 Test vessels

As test vessels, 400 ml glass beakers containing approximately 250 ml of test solution were used. They were covered with glass plates during the test.

3.5 Test room and light regime

The test was carried out in a temperature-controlled room with a light regime of 16 h of ambient light per day, provided by fluorescent tubes.

3.6 Test animals

The test was performed with *Daphnia magna* (water fleas), of which a continuous culture is maintained at Akzo Nobel Chemicals Research, Arnhem, Dept. CGS-ENV according to Standard Operation Procedure E 1 (8.4). The animals used in the test were less than 24 hours old at the beginning of the test and were obtained from parent animals having an age of 2-4 weeks.

4. METHODS

4.1 Test principle and procedures

The test was performed as a static test. The total duration was 48 hours. 20 daphnids divided into 4 batches of 5 animals were used per test concentration and control

Under otherwise identical test conditions, the daphnids were exposed to the chosen concentrations of the test substance as described below and immobility and sub-lethal effects were recorded at approximately 24 and 48 hours. The daphnids were considered immobile when they were not able to swim for 15 seconds after gentle agitation of the test vessel. In addition to immobility, sub-lethal effects such as floating at the surface were recorded.

The daphnids were randomly placed in the test solutions and the test vessels were positioned in a random manner. During the test the vessels were covered with glass plates. The test solutions were not aerated. The animals were not fed during the test.

4.2 Preparation of the test solutions

The test solution was a Water Accommodated Fraction (WAF) prepared from 1.01 g test substance in 2 litres of DSW. This solution was slowly stirred for approximately 48 hours, then transferred into a separation funnel and left at test temperature for phase separation. After approximately 3 hours, when phase separation was complete, the aqueous phase was withdrawn and used to prepare the following dilutions: 1:16 – 1:8 – 1:4 – 1:2 and the undiluted WAF. A control containing only test medium was included in the test.

4.3 Preparation of the test medium

As test medium so called Dutch Standard water was used, with a pH of approximately 8.2 and a hardness of approximately 12°dH, containing per liter of deionized water: 100 mg of $NaHCO_3$, 20 mg of $KHCO_3$, 200 mg of $CaCl_2 \cdot 2H_2O$ and 180 mg of $MgSO_4 \cdot 7H_2O$ (Standard Operation Procedure E 1(8.4)). The test

medium was aerated before being used in the test. The air was water-saturated and purified by an active coal and cotton filter.

4.4 Chemical analyses.

At the start of the test samples of about 15 ml were taken from the test vessels with the undiluted WAF and the control, just before the distribution of the test solutions over the four batches and adding the test animals. At the end of the test the solutions from the four batches were combined and mixed and 15 ml samples from the undiluted WAF and the control were used for analysis. Samples were stored in a refrigerator until analyses. Chemical analysis of the test concentrations were performed according to the analytical procedure described in Annex 2

4.5 Determination of dissolved oxygen, pH and temperature in the test solutions

The dissolved oxygen and pH of all test concentrations and controls were measured at the beginning and at the end of the test. The temperature was measured continuously in an additional test vessel filled with deionized water and kept under identical conditions as the actual test vessels. The dissolved oxygen concentrations were determined electrochemically using an oxygen electrode and meter according to Standard Operation Procedure K 2 (8.5). The pH was measured using a microcomputer pH-meter according to Standard Operation Procedure K 1 (8.6). The temperature was measured with a recorder and thermo-couple according to Standard Operation Procedure K 6 (8.7).

4.6 Evaluation of data

The EC_{50} was calculated with the computer program TOXCALC™ version 5.0 according to Standard Operation Procedure L 2, using the trimmed Spearman-Kärber method. The concentration as measured in the WAF at the beginning of the test was used to calculate the concentrations of the dilutions. The concentrations causing zero and 100% immobility were derived directly from the test observations.

5. RESULTS

5.1 Toxicity

The results of the test on mobility and the results of the statistical evaluation of the data are summarized in Tables 1 and 2.

The EC_{50} - 48 h was calculated to be 0.79 mg/l with 95% confidence limits of 0.68 and 0.93 mg/l, the EC_{50} - 24h was 1.16 mg/l.

The NOEC 48 h amounted to 0.381 mg/l, the LOEC 48 h was 0.763 mg/l. The EC_{100} was 1.525 mg/l and was observed after 48 h.

Sublethal effects were not observed in the test concentrations where no immobility was observed.

5.2 Chemical analyses

The analyses were all performed in compliance with the validity criteria as stated in the analytical procedure included in the study plan and in Annex 2. All samples were analyzed in duplicate. One chromatogram of a control sample was however slightly disturbed by an electronic pulse. The remaining peaks on the chromatogram were used for interpretation and were identical with the duplicate. The duplicate with the undisturbed chromatogram was used for interpretation of the concentration in the sample.
The results of the analyses are presented in table 5.
The results show that the undiluted WAF contains 3.05 mg/l tert-butyl peroxyneodecanoate at the beginning of the test and below the detection limit (1.64 mg/l) at the end of the test, which is less than 80% of the initial concentration. These results show that the test substance is probably not stable under test conditions. The concentration of the WAF at the beginning of the test was used for the calculation of the toxicity.

5.3 Oxygen, pH and temperature

The maximum variation in oxygen concentration during the test was 0.5 mg/l.
The results of the oxygen measurements are summarized in Table 3.

The maximum variation of pH observed during the test was 0.6 units.
The results of the pH measurements are summarized in Table 4.

The temperature in the test vessels during the test period ranged from 19.3 to 21.0°C.

6. QUALITY CRITERIA

The following quality criteria have been met in the present study:

- Immobilization in the control did not exceed 10% at the end of the test.
- The oxygen concentration was not less than 2 mg/l at the end of the test.
- There were no test animals trapped at the surface of the water in the control group.

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7. DEVIATIONS FROM THE STUDY PLAN

- The measured concentrations of the test substance in the WAF at the end of the test were lower than 80% of the measured concentration at the beginning of the test.

8. REFERENCES

8.1 OECD Guidelines for testing of chemicals, 202; (1984.04.04)

8.2 EEC directive 79/831, Annex V, part C.: Methods for the determination of ecotoxicity, part C.2. Acute toxicity to Daphnia (updated version 11/1989)

8.3 Standard Operation Procedure K 10: Deionizer

8.4 Standard Operation Procedure E 1: Culturing of Daphnia magna

8.5 Standard Operation Procedure K 2: Oxygen meters

8.6 Standard Operation Procedure K 1: pH-meter

8.7 Standard Operation Procedure K 6: Thermometers

8.8 Standard Operation Procedure L 2: Acute toxicity test of fish and daphnia magna (calculation of effects with the TOXCALC™ computer program)

8.9 M.A. Hamilton, P.C. Russo and R.V. Thurston, 1977: 'Trimmed Spearman-Kärber method for estimating median lethal concentrations in toxicity bioassays'. Env.Sci. & Technol. 11, 714-719. Correction, 12 (1978) 417

8.10 C.E. Stephan, 1977: 'Methods for calculating an LC_{50}'. In: Aquatic toxicology and hazard evaluation. F.L. Mayer and J.L. Hamelink (Eds.), 65-84

Table 1
Test results

Test substance (mg/l)	Batches	Number of mobile animals		
		0 hours	24 hours	48 hours
Control	Batch I	5	5	5
	Batch II	5	5	5
	Batch III	5	5	4
	Batch IV	5	5	5
	Total	20	20	19
0.1906	Batch I	5	5	5
	Batch II	5	5	5
	Batch III	5	5	5
	Batch IV	5	5	5
	Total	20	20	20
0.3813	Batch I	5	5	5
	Batch II	5	5	5
	Batch III	5	5	5
	Batch IV	5	5	5
	Total	20	20	20
0.7625	Batch I	5	5	1
	Batch II	5	5	4
	Batch III	5	5	4
	Batch IV	5	5	2
	Total	20	20	11
1.525	Batch I	5	0	0
	Batch II	5	0	0
	Batch III	5	2	0
	Batch IV	5	0	0
	Total	20	2	0
3.05	Batch I	5	0	0
	Batch II	5	0	0
	Batch III	5	0	0
	Batch IV	5	0	0
	Total	20	0	0

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Table 2
Statistical evaluation

Time (hours)	EC_{50} (mg/l)	95% confidence limits (mg/l)
24	1.16	1.05 - 1.27
48	0.79	0.68 - 0.93

Table 3
Oxygen measurements

Dilution (mg/l)	0	48 h
Control	8.5	9.0
0.1906	8.5	8.9
0.3813	8.6	8.9
0.7625	8.6	9.0
1.525	8.6	8.9
3.05	8.5	8.9

Table 4
pH-measurements

Dilution (mg/l)	0	48 h
Control	8.1	8.0
0.1906	8.1	8.0
0.3813	8.0	8.0
0.7625	7.9	8.1
1.525	7.8	8.1
3.05	7.6	8.1

Table 5
Chemical analyses

Sample	0h	48 h
Control (mg/l)	0	0
Undiluted WAF (mg/l)	3.05	1*

* = Concentration was below the limit of detection (= 1.64 mg/l)

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Annex 1

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Certificate of analysis

Chemicals

Delivery address

AKZO NOBEL CHEMICALS RESEARCH
R.F. VAN WIJK
VELPERWEG
ARNHEM
THE NETHERLANDS

Page 1 of 1

Our ref. ICS 331 Ghlin date 10.01.2001

Order number Akzo Nobel : 8137001799 Your ref :
Product name : Trigonox 23-C75
Product code : 65836 Delivery No : 8132023658
Total Batches : 1
Total Quantity : 0.05 kg Delivery date : 10.01.2001

Packages : 1
Quantity : 0.05 kg

Analysis of	Unit	Results	Specification	Test Method
Lot / Batch 00101130675				
Colour	Pt-Co	5	0 - 50	Col/84.3
Assay	%	75.0	74.0 - 78.0	Jo/72.13
TBHP	mg/kg	318	0 - 1000	GC/79.2
Inorg. +org.hydr chloride	mg/kg	21.00	0 - 150	Ag/90.1

For method of analysis an equivalent test method may have been used.

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S.A. Akzo Nobel Chemicals N.V.
Works
Parc Industriel de Chim Zone A
B-7011
Belgique

Tel. +32(0)65 642381

Fax +32(0)65 642383

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Annex 2

Description of the analytical procedure for tert-butyl peroxyneodecanoate in solvent

1. Introduction

A method is described to determine the concentration of the test substance in water by U.V. detection at 215 nm. Procedures and instrumentation were based on HPLC.

2. Analytical procedure

The following conditions were found to be suitable for the determination of the test compound for concentrations of 0 to 40 mg/l in deionized water.

- Autosampler: Spark model marathon equipped with a 50 µl sample loop
- Column: Waters, Symmetry C18 5 µm; 4.6 x 150 mm cartridge column and a Waters Symmetry C18 guard column
- Mobile Phase: methanol : water = 85 : 15 v/v%
- Detector: Separations UV/VIS detector model 759A
- Recorder: Kipp &Zn, 10 mV
- Integrator: Chromatography server, Atlas 2000 release 1
- Pump: Separations model 300 HPLC pump
- Eluent flow rate: 0.5 ml/min
- Detection at: 215 nm

A stock solution of the test compound was made in mobile phase for HPLC analyses. At the beginning the analysis series, calibration standards were analyzed (n=5). HPLC-analyses were performed in duplicate. Samples were injected directly into the HPLC-system. Control standards from the middle range of the calibration series were analyzed at a minimum rate of one per ten samples.

3 Calculation of concentrations

Quantification was done by measurement of peak areas. The concentration of the test substance in the samples was calculated from the relation between concentration of standards (Cs) and peak area (PAs) obtained with linear regression analysis:

PAs = Cs * Slope + constant

$$\text{Sample concentration} = \frac{\text{Sample peak area} - \text{constant}}{\text{slope}}$$

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4 Reproducibility and validation

With the system described above the test substance elutes after about 13.5 minutes.
The analytical method was found to be linear over the range of 0 to 17 mg/l of standard solutions of test substance using the conditions described above. The HPLC calibration analyses gave a linear regression with a squared regression coefficient $r^2 \geq 0.99$ (n=5). Control standards analyzed during the analyses was within 2 % of the expected values based on the calibration curve.

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Distribution

Clients

Akzo Nobel Polymer Chemicals, E. Houthoff
P.O. Box 247
3800 AE Amersfoort
The Netherlands

Laporte GmbH,
Dr. GustavsonAdolphstrasse 3
82049 Pullach
Germany

Atofina
Cours Michelet
La Defense 10
92091 Paris
France

Others

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CGS-ENV/Geurts, GLP-archive
Central file (1x)

Abstract

Research Center Manager

CRM/Verhelst